

Chemical Distribution Analysis by Micro X-ray Fluorescence

Introduction

There is a need for analyzing the elemental distribution of a sample, across a line, or within a specified area. Micro X-ray Fluorescence (Micro-XRF) provides fast, non-destructive elemental analysis at a point, line or specified area. In the example shown, EDAX's Orbis Micro-XRF Elemental Analyzer is used to quantify a Manganese (Mn)-rich inclusion in a steel. The Orbis Micro-XRF Elemental Analyzer performs chemical distribution analysis by combining capillary optics to concentrate the X-ray beam to a small area, with an automated three-axis stage to move the sample to required trajectories. Element distributions (chemical concentration profiles) can be obtained by moving the sample under the X-ray beam.

Samples

In this study, a stainless steel sample with a high manganese (Mn) concentration inclusion was profiled by effectively scanning the probe beam across the region of interest. The inclusion is optically visible as the light stripe in Figure 1.



Figure 1. An overview of the sample at 10X magnification.

Figure 2 shows the inclusion at higher magnification. The horizontal line represents the area scanned and the circle at the center represents the X-ray excitation area. A micron marker is also shown for reference.

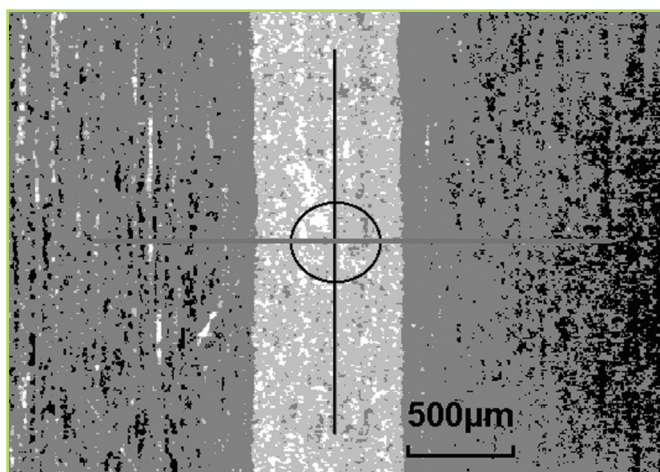


Figure 2. A detailed picture of the sample at 100X magnification.

Measuring Conditions

Measurements were made using a capillary optic system for X-ray excitation. Table 1 lists the analytical conditions used.

Excitation	
Tube	Mo-Target
Power	40 kV, 120µA
Capillary Diameter	300 µm
Measurement	
Detector	30 mm ² SDD
Window	Be
Resolution	≤ 135 eV, Mn-Kα
Step Size	300 µm
Dwell time/point	5 live seconds

Table 1.

Results

The spectra were obtained and correlated to the measuring points and quantification was done using standardless analysis. Figure 3 shows a spectral comparison of normal steel (bulk) versus the higher Mn inclusion.

The spectrum in Figure 3 exhibits the iron (Fe) intensity to be relatively constant between bulk and inclusion. However, the difference in net Mn intensity is clearly evident and exhibits a difference in concentration by nearly a factor of two: from 2% (bulk) to 4% (inclusion) when quantified. The escape peak of the high concentration Fe is also exhibited. Continuing the analysis by plotting the concentration as a function of beam position produces the distribution exhibited in Figure 4. Clearly, the higher Mn concentration correlates well with the lighter stripe running through the sample.

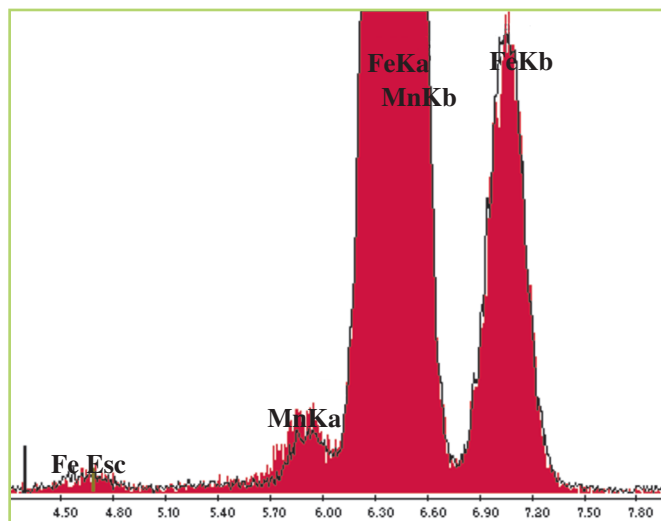


Figure 3. Comparison of Mn-enriched inclusion (solid red) and bulk steel (black outline).

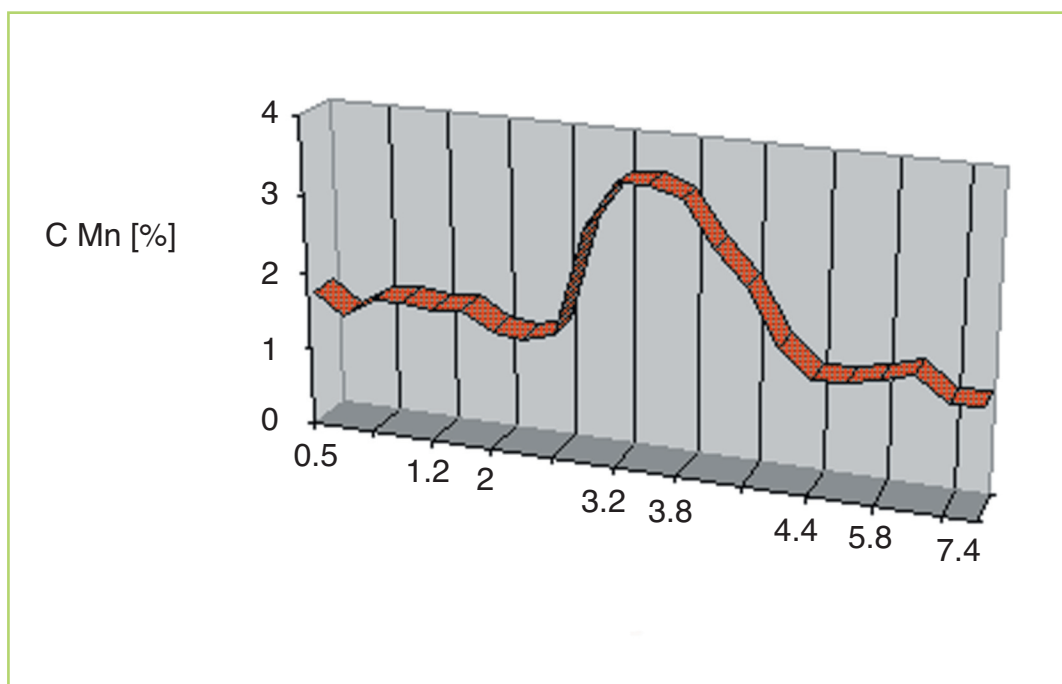


Figure 4. Distribution of Mn concentration.

Conclusion

The Orbis Micro-XRF Elemental Analyzer has been used to quantify a Mn-rich inclusion in a steel. In this measurement example, the variation in Mn wt% was from 2 to 4 wt% and was easily measured with statistical significance at 5 live seconds per point. Increased tube power or longer measuring times would allow analysis of Mn-rich inclusions with smaller variations in wt% with respect to the base steel.